Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,1,3-Trimethyl-3-(4-nitrophenyl)indane

Jian Men, Shi-Xu Yi, Fang Bo, Hua Chen and Guo-Wei Gao*

College of Chemistry, Sichuan University, Chengdu 610064, People's Republic of China
Correspondence e-mail: gaosunday@yahoo.com.cn

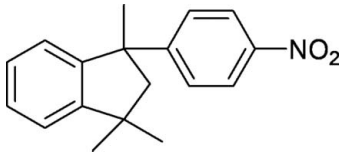
Received 21 July 2008; accepted 28 August 2008

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.051; wR factor = 0.148; data-to-parameter ratio = 14.3.

In the title compound, $C_{18}H_{19}NO_2$, the five-membered ring of the indane fragment adopts an envelope conformation, with the unsubstituted C atom, acting as the flap atom, deviating by 0.412 (3) Å from the plane through the remaining four atoms. The dihedral angle between the nitrophenyl ring and the indane benzene ring is 72.5 (1)°. The distances from the two O atoms to the plane of the adjacent benzene ring are 0.113 (4) and 0.064 (4) Å.

Related literature

For related literature, see: Bateman & Gordon (1976); Bezdek & Hrabak *et al.* (1979); Kumar *et al.* (1983); Men *et al.* (2008); Hanaineh-Abdelnour *et al.* (1999).



Experimental

Crystal data

$C_{18}H_{19}NO_2$ M_r = 281.34
Monoclinic, $P2_1/c$
a = 11.305 (4) Å
b = 11.422 (2) Å
c = 11.963 (2) Å
β = 102.32 (4)°
V = 1509.1 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 294 (2) K
0.48 × 0.42 × 0.40 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
3836 measured reflections
2808 independent reflections
1569 reflections with $I > 2\sigma(I)$
R_{int} = 0.005
3 standard reflections every 200 reflections
intensity decay: 0.3%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.148
S = 1.02
2808 reflections
197 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Data collection: *DIFRAC* (Gabe & White, 1993); cell refinement: *DIFRAC*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The authors are grateful to the National Undergraduates' Innovative Experiment Project of China for financial support and thank Mr Zhi-Hua Mao of Sichuan University for the X-ray data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KJ2096).

References

Bateman, J. & Gordon, D. A. (1976). US Patent 3 983 092.
Bezdek, M. & Hrabak, F. J. (1979). *Polym. Sci. Polym. Chem. Ed.* **17**, 2857.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
Gabe, E. J. & White, P. S. (1993). American Crystallographic Association Pittsburgh Meeting, Abstract PA104.
Hanaineh-Abdelnour, L., Bayyuk, S. & Theodorie, R. (1999). *Tetrahedron*, **50**, 11859–11870.
Kumar, D., Fohlen, G. M. & Parker, J. A. (1983). *Macromol. Chem.* **16**, 1250–1257.
Men, J., Yang, M.-J., Jiang, Y., Chen, H. & Gao, G.-W. (2008). *Acta Cryst.* E**64**, o847.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2008). E**64**, o2192 [**doi:10.1107/S1600536808027633**]

1,1,3-Trimethyl-3-(4-nitrophenyl)indane

J. Men, S.-X. Yi, F. Bo, H. Chen and G.-W. Gao

Comment

Maleimide and its substituted derivatives are well known monomers that have many applications in industry. It may be used to prepare heat resistant polymers or copolymers (Kumar *et al.*, 1983; Bezdek & Hrabak *et al.*, 1979). Excellent thermal properties of the maleimide polymers and copolymers have also attracted much attention (Hanaineh-Abdelnour *et al.*, 1999). The title compound is an important intermediate for synthesis of maleimide and its substituted derivatives. (Bateman & Gordon, 1976). Phenylindane amines is prepared by a process comprising acid-catalyzed dimerization of α-methylstyrene and subsequent nitration and reduction of the 1,1,3-trimethyl-3-phenyl-2,3-dihydro-1*H*-indene (Bateman & Gordon, 1976).

In the molecule of the title compound (Fig.1), the bond lengths and angles of the phenylidane moiety are comparable with those observed in 1,1,3,-trimethyl-3-phenyl-2,3-dihydro-1*H*-indene (Men *et al.*, 2008). Ring A (C1—C6) and B (C13—C18) are planar and have a dihedral angle of 72.5 (1)°. The B ring forms a dihedral angle of 27.1 (3)° with the plane defined by the indane Csp3 atoms C7, C9 and C10. The torsion angles O1—N1—C3—C2 and O2—N1—C3—C4 are -174.3 (2)° and -176.5 (2)°, respectively. The distances of the O atoms to the plane through the adjacent benzene ring are 0.113 (4) Å and 0.064 (4) Å, respectively. The five-membered ring of the indane fragment adopts an envelop conformation, with the unsubstituted C atom acting as the flag atom, deviating 0.412 (3) Å from the plane through the remaining four atoms.

Experimental

To a three-necked, 250 ml flask equipped with a mechanical stirrer, 23.6 g (0.1 mol) 1,1,3-trimethyl-3-phenylindane, dissolved in 75 ml chloroform, was added. The flask was placed in an ice bath at 273 K. A previously mixed acidic solution containing 39.6 ml H_2SO_4 and 13.2 ml HNO_3 was added dropwise to the phenylidane solution over 4 h at 273 K. The chloroform phase was isolated and washed with an aqueous bicarbonate solution and then with distilled water until neutral. The chloroform was removed with a rotovaporator, thereby yielding a viscous yellow liquid, which was recrystallized from methanol, afforded a light yellow powder (22.0 g, yield 78%, m.p.452–455 K). Single crystals suitable for X-ray diffraction were obtained at room temperature by slow evaporation of ethyl acetate over a period of several days.

Refinement

H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined with a riding model (U =0.06688–0.08804 Å^2)

Figures

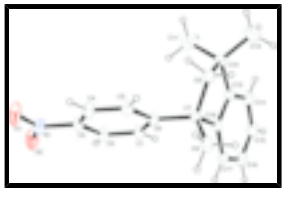


Fig. 1. The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level.

1,1,3-Trimethyl-3-(4-nitrophenyl)indane

Crystal data

$C_{18}H_{19}NO_2$	$F_{000} = 600$
$M_r = 281.34$	$D_x = 1.238$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 11.305$ (4) Å	Cell parameters from 25 reflections
$b = 11.422$ (2) Å	$\theta = 4.6$–7.5º
$c = 11.963$ (2) Å	$\mu = 0.08$ mm^{-1}
$\beta = 102.32$ (4)º	$T = 294$ (2) K
$V = 1509.1$ (7) Å^3	Block, colourless
$Z = 4$	0.48 × 0.42 × 0.40 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.005$
Radiation source: fine-focus sealed tube	$\theta_{max} = 25.6$º
Monochromator: graphite	$\theta_{min} = 1.8$º
$T = 294$(2) K	$h = -13\rightarrow13$
$\omega/2\theta$ scans	$k = 0\rightarrow13$
Absorption correction: none	$l = -9\rightarrow14$
3836 measured reflections	3 standard reflections
2808 independent reflections	every 200 reflections
1569 reflections with $I > 2\sigma(I)$	intensity decay: 0.3%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.050$	$w = 1/[\sigma^2(F_o^2) + (0.0825P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.148$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.02$	$\Delta\rho_{max} = 0.20$ e Å^{-3}
2808 reflections	$\Delta\rho_{min} = -0.18$ e Å^{-3}
197 parameters	Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.017 (3)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.3433 (2)	−0.30042 (15)	0.44243 (16)	0.0958 (7)
O2	0.3595 (2)	−0.17051 (16)	0.31987 (16)	0.0899 (7)
N1	0.34612 (19)	−0.19883 (17)	0.41338 (17)	0.0587 (6)
C1	0.3077 (2)	0.08995 (18)	0.53719 (17)	0.0510 (6)
H1	0.3021	0.1679	0.5142	0.067 (3)*
C2	0.3234 (2)	0.00613 (19)	0.45991 (18)	0.0525 (6)
H2	0.3287	0.0263	0.3858	0.067 (3)*
C3	0.3312 (2)	−0.10856 (17)	0.49490 (17)	0.0454 (5)
C4	0.3226 (2)	−0.13981 (18)	0.60329 (18)	0.0523 (6)
H4	0.3265	−0.2181	0.6251	0.067 (3)*
C5	0.3082 (2)	−0.05412 (19)	0.67899 (17)	0.0521 (6)
H5	0.3038	−0.0750	0.7531	0.067 (3)*
C6	0.29992 (18)	0.06319 (18)	0.64821 (16)	0.0412 (5)
C7	0.2858 (2)	0.15488 (17)	0.73769 (16)	0.0447 (5)
C8	0.3994 (2)	0.1497 (2)	0.83509 (18)	0.0616 (7)
H8A	0.4704	0.1586	0.8039	0.088 (3)*
H8B	0.4022	0.0756	0.8735	0.088 (3)*
H8C	0.3963	0.2117	0.8886	0.088 (3)*
C9	0.1683 (2)	0.13740 (19)	0.78272 (18)	0.0544 (6)
H9A	0.1440	0.0558	0.7758	0.068 (5)*
H9B	0.1819	0.1594	0.8628	0.068 (5)*
C10	0.0684 (2)	0.21467 (19)	0.71127 (19)	0.0546 (6)
C11	−0.0054 (3)	0.1487 (2)	0.6080 (2)	0.0786 (8)
H11A	−0.0597	0.2022	0.5606	0.088 (3)*
H11B	−0.0513	0.0877	0.6342	0.088 (3)*
H11C	0.0483	0.1151	0.5646	0.088 (3)*
C12	−0.0166 (3)	0.2629 (3)	0.7825 (3)	0.0865 (9)
H12A	0.0289	0.3071	0.8455	0.088 (3)*
H12B	−0.0570	0.1992	0.8113	0.088 (3)*
H12C	−0.0756	0.3127	0.7359	0.088 (3)*
C13	0.1449 (2)	0.30897 (18)	0.67178 (18)	0.0479 (6)
C14	0.1073 (3)	0.4174 (2)	0.6255 (2)	0.0638 (7)
H14	0.0267	0.4399	0.6150	0.067 (3)*

C15	0.1915 (3)	0.4909 (2)	0.5954 (2)	0.0656 (7)
H15	0.1672	0.5637	0.5640	0.067 (3)*
C16	0.3109 (3)	0.4589 (2)	0.6106 (2)	0.0629 (7)
H16	0.3663	0.5101	0.5896	0.067 (3)*
C17	0.3492 (2)	0.3515 (2)	0.65694 (19)	0.0539 (6)
H17	0.4302	0.3298	0.6680	0.067 (3)*
C18	0.2645 (2)	0.27631 (17)	0.68677 (16)	0.0437 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.170 (2)	0.0401 (10)	0.0774 (12)	0.0122 (11)	0.0259 (13)	−0.0062 (9)
O2	0.152 (2)	0.0637 (11)	0.0666 (12)	0.0012 (12)	0.0525 (12)	−0.0106 (9)
N1	0.0753 (15)	0.0474 (12)	0.0535 (12)	0.0026 (10)	0.0136 (10)	−0.0071 (9)
C1	0.0736 (18)	0.0365 (11)	0.0463 (11)	0.0024 (11)	0.0204 (11)	0.0039 (9)
C2	0.0739 (17)	0.0448 (13)	0.0426 (11)	−0.0010 (11)	0.0208 (11)	0.0024 (10)
C3	0.0503 (15)	0.0411 (11)	0.0457 (11)	0.0029 (10)	0.0124 (10)	−0.0051 (9)
C4	0.0687 (17)	0.0375 (12)	0.0505 (13)	0.0069 (11)	0.0124 (11)	0.0045 (9)
C5	0.0695 (17)	0.0468 (13)	0.0414 (11)	0.0073 (11)	0.0152 (11)	0.0075 (9)
C6	0.0439 (13)	0.0387 (11)	0.0424 (11)	0.0003 (10)	0.0126 (9)	0.0014 (9)
C7	0.0515 (14)	0.0420 (11)	0.0419 (11)	0.0028 (10)	0.0131 (10)	−0.0008 (9)
C8	0.0662 (17)	0.0681 (16)	0.0482 (12)	0.0069 (13)	0.0069 (11)	−0.0068 (11)
C9	0.0644 (17)	0.0517 (14)	0.0530 (12)	0.0029 (12)	0.0258 (11)	0.0020 (10)
C10	0.0490 (15)	0.0538 (14)	0.0648 (14)	−0.0004 (11)	0.0208 (12)	0.0004 (11)
C11	0.0667 (19)	0.0772 (19)	0.0885 (19)	−0.0178 (16)	0.0091 (15)	0.0037 (15)
C12	0.080 (2)	0.081 (2)	0.113 (2)	0.0119 (17)	0.0548 (19)	0.0061 (17)
C13	0.0481 (15)	0.0444 (12)	0.0532 (12)	0.0030 (11)	0.0152 (10)	−0.0057 (10)
C14	0.0608 (17)	0.0524 (15)	0.0776 (16)	0.0115 (13)	0.0135 (13)	0.0005 (12)
C15	0.083 (2)	0.0401 (13)	0.0751 (16)	0.0022 (14)	0.0209 (15)	0.0023 (11)
C16	0.082 (2)	0.0423 (14)	0.0683 (15)	−0.0165 (13)	0.0241 (14)	−0.0082 (11)
C17	0.0547 (16)	0.0492 (13)	0.0595 (13)	−0.0078 (12)	0.0160 (11)	−0.0112 (11)
C18	0.0497 (15)	0.0397 (12)	0.0439 (11)	−0.0010 (10)	0.0147 (10)	−0.0082 (9)

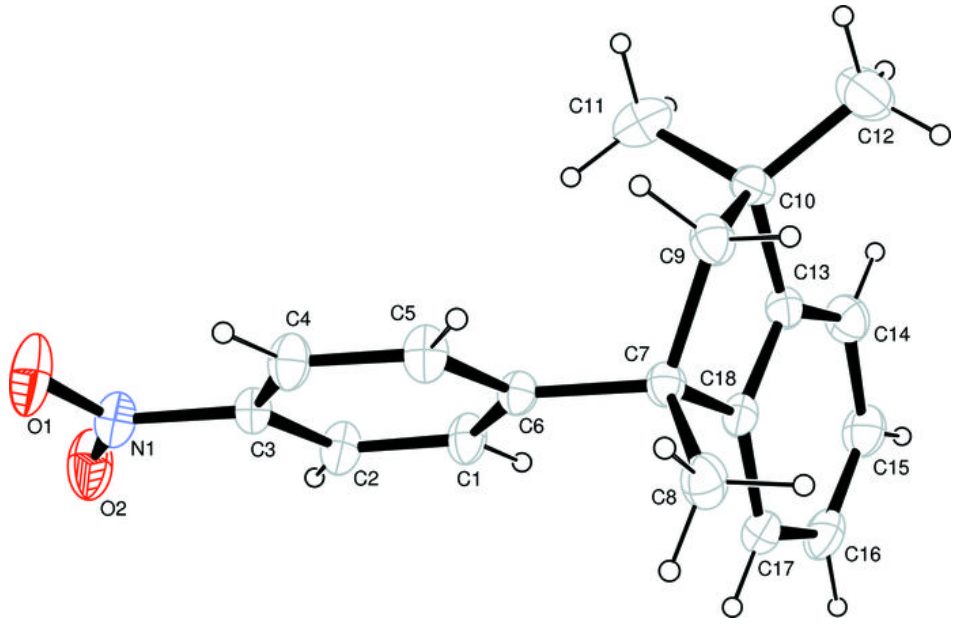
Geometric parameters (Å, °)

O1—N1	1.214 (2)	C9—H9A	0.9700
O2—N1	1.205 (2)	C9—H9B	0.9700
N1—C3	1.454 (3)	C10—C12	1.518 (3)
C1—C2	1.368 (3)	C10—C13	1.518 (3)
C1—C6	1.384 (3)	C10—C11	1.533 (3)
C1—H1	0.9300	C11—H11A	0.9600
C2—C3	1.372 (3)	C11—H11B	0.9600
C2—H2	0.9300	C11—H11C	0.9600
C3—C4	1.368 (3)	C12—H12A	0.9600
C4—C5	1.366 (3)	C12—H12B	0.9600
C4—H4	0.9300	C12—H12C	0.9600
C5—C6	1.387 (3)	C13—C18	1.377 (3)
C5—H5	0.9300	C13—C14	1.385 (3)
C6—C7	1.530 (3)	C14—C15	1.374 (4)

C7—C18	1.513 (3)	C14—H14	0.9300
C7—C8	1.540 (3)	C15—C16	1.372 (3)
C7—C9	1.550 (3)	C15—H15	0.9300
C8—H8A	0.9600	C16—C17	1.377 (3)
C8—H8B	0.9600	C16—H16	0.9300
C8—H8C	0.9600	C17—C18	1.389 (3)
C9—C10	1.540 (3)	C17—H17	0.9300
O2—N1—O1	122.6 (2)	H9A—C9—H9B	108.4
O2—N1—C3	119.22 (19)	C12—C10—C13	112.8 (2)
O1—N1—C3	118.2 (2)	C12—C10—C11	109.2 (2)
C2—C1—C6	122.54 (19)	C13—C10—C11	110.26 (19)
C2—C1—H1	118.7	C12—C10—C9	111.9 (2)
C6—C1—H1	118.7	C13—C10—C9	100.42 (18)
C1—C2—C3	118.08 (19)	C11—C10—C9	112.0 (2)
C1—C2—H2	121.0	C10—C11—H11A	109.5
C3—C2—H2	121.0	C10—C11—H11B	109.5
C4—C3—C2	121.69 (19)	H11A—C11—H11B	109.5
C4—C3—N1	119.52 (19)	C10—C11—H11C	109.5
C2—C3—N1	118.78 (19)	H11A—C11—H11C	109.5
C5—C4—C3	118.9 (2)	H11B—C11—H11C	109.5
C5—C4—H4	120.5	C10—C12—H12A	109.5
C3—C4—H4	120.5	C10—C12—H12B	109.5
C4—C5—C6	121.78 (19)	H12A—C12—H12B	109.5
C4—C5—H5	119.1	C10—C12—H12C	109.5
C6—C5—H5	119.1	H12A—C12—H12C	109.5
C1—C6—C5	116.97 (18)	H12B—C12—H12C	109.5
C1—C6—C7	123.90 (18)	C18—C13—C14	120.2 (2)
C5—C6—C7	119.12 (17)	C18—C13—C10	112.06 (18)
C18—C7—C6	112.16 (15)	C14—C13—C10	127.8 (2)
C18—C7—C8	112.04 (18)	C15—C14—C13	118.7 (3)
C6—C7—C8	107.99 (17)	C15—C14—H14	120.6
C18—C7—C9	100.55 (17)	C13—C14—H14	120.6
C6—C7—C9	112.34 (17)	C16—C15—C14	121.3 (2)
C8—C7—C9	111.73 (18)	C16—C15—H15	119.4
C7—C8—H8A	109.5	C14—C15—H15	119.4
C7—C8—H8B	109.5	C15—C16—C17	120.4 (2)
H8A—C8—H8B	109.5	C15—C16—H16	119.8
C7—C8—H8C	109.5	C17—C16—H16	119.8
H8A—C8—H8C	109.5	C16—C17—C18	118.7 (2)
H8B—C8—H8C	109.5	C16—C17—H17	120.7
C10—C9—C7	108.33 (17)	C18—C17—H17	120.7
C10—C9—H9A	110.0	C13—C18—C17	120.7 (2)
C7—C9—H9A	110.0	C13—C18—C7	111.63 (19)
C10—C9—H9B	110.0	C17—C18—C7	127.7 (2)
C7—C9—H9B	110.0		
C6—C1—C2—C3	0.2 (4)	C7—C9—C10—C11	91.7 (2)
C1—C2—C3—C4	0.5 (4)	C12—C10—C13—C18	134.8 (2)
C1—C2—C3—N1	179.2 (2)	C11—C10—C13—C18	−102.8 (2)

O2—N1—C3—C4	−176.5 (2)	C9—C10—C13—C18	15.5 (2)
O1—N1—C3—C4	4.4 (3)	C12—C10—C13—C14	−45.1 (3)
O2—N1—C3—C2	4.9 (3)	C11—C10—C13—C14	77.3 (3)
O1—N1—C3—C2	−174.3 (2)	C9—C10—C13—C14	−164.4 (2)
C2—C3—C4—C5	−1.2 (4)	C18—C13—C14—C15	−0.1 (3)
N1—C3—C4—C5	−179.8 (2)	C10—C13—C14—C15	179.7 (2)
C3—C4—C5—C6	1.2 (3)	C13—C14—C15—C16	−0.2 (4)
C2—C1—C6—C5	−0.3 (3)	C14—C15—C16—C17	0.0 (4)
C2—C1—C6—C7	178.3 (2)	C15—C16—C17—C18	0.5 (3)
C4—C5—C6—C1	−0.4 (3)	C14—C13—C18—C17	0.6 (3)
C4—C5—C6—C7	−179.1 (2)	C10—C13—C18—C17	−179.25 (18)
C1—C6—C7—C18	7.7 (3)	C14—C13—C18—C7	−179.66 (18)
C5—C6—C7—C18	−173.75 (19)	C10—C13—C18—C7	0.4 (2)
C1—C6—C7—C8	−116.2 (2)	C16—C17—C18—C13	−0.8 (3)
C5—C6—C7—C8	62.3 (3)	C16—C17—C18—C7	179.52 (19)
C1—C6—C7—C9	120.1 (2)	C6—C7—C18—C13	103.5 (2)
C5—C6—C7—C9	−61.4 (3)	C8—C7—C18—C13	−134.8 (2)
C18—C7—C9—C10	25.7 (2)	C9—C7—C18—C13	−16.0 (2)
C6—C7—C9—C10	−93.8 (2)	C6—C7—C18—C17	−76.8 (3)
C8—C7—C9—C10	144.68 (18)	C8—C7—C18—C17	44.8 (3)
C7—C9—C10—C12	−145.3 (2)	C9—C7—C18—C17	163.6 (2)
C7—C9—C10—C13	−25.3 (2)		

Fig. 1


C11
C12
C10
C9
C13
C14
C4
C5
C7
C18
O1
N1
C3
C6
C15
C2
C1
C8
C17
C16
O2